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Bray, R., Barton, J., Madsen, D., & van Hees, P. (2022). Burning material behaviour in hypoxic environments: An experimental study examining a representative storage arrangement of acrylonitrile butadiene styrene, polyethylene bubble wrap, and cardboard layers as a composite system. *Fire and Materials*, 46(1), 313-328. <https://doi.org/10.1002/fam.3002>

[Link to publication record in Ulster University Research Portal](#)

**Published in:**  
Fire and Materials

**Publication Status:**  
Published (in print/issue): 01/01/2022

**DOI:**  
[10.1002/fam.3002](https://doi.org/10.1002/fam.3002)

**Document Version**  
Publisher's PDF, also known as Version of record

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## RESEARCH ARTICLE

# Burning material behaviour in hypoxic environments: An experimental study examining a representative storage arrangement of acrylonitrile butadiene styrene, polyethylene bubble wrap, and cardboard layers as a composite system

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## Summary

Cone calorimeter and controlled atmosphere cone calorimeter experiments were conducted on various samples. The intent of the tests was to examine the behavior of uniform and composite samples in a range of thicknesses, irradiances, and oxygen concentrations. Single, uniform layers of acrylonitrile butadiene styrene (ABS) were compared to a composite mix, comprising of ABS with a surface layer of cardboard and a secondary layer of polyethylene bubble wrap (intended to represent a potential storage arrangement). The horizontal samples have been tested at irradiances of 25 and 50 kW/m<sup>2</sup> and oxygen concentrations of 20.95%, 17%, and 15% to examine a range of significant variables. Results for the uniform arrangement indicated various correlations, previously observed in the works of others, such as the relationships typically described between applied heat flux, ignitability, heat release rate and the effect of the introduction of hypoxic conditions. However, results were shown to change significantly when samples were arranged to feature composite layers. A hypothesized cause of the behavioral change, namely the soot and char residual introduced from the incomplete combustion of the cardboard layer, highlights further important variables that require consideration in material testing under hypoxic conditions. Such variables, namely specific material behaviors and sample orientation, must be sufficiently captured in the design methodologies of systems reliant upon the introduction of hypoxic conditions. It is concluded that sufficiently capturing a wider range of variables in burning materials under hypoxic conditions will introduce further design resilience and help optimize fire protection/prevention methods.

## KEYWORDS

ABS, bubble wrap, cardboard, composite, cone calorimetry, hypoxic, ORS, polymer

## 1 | INTRODUCTION

There is a limited body of knowledge within the fire research community concerning the study of materials formed of composite layers,

particularly when exposed to hypoxic conditions.<sup>1</sup> While there is more data available observing the behavior of materials of a uniform composition under fire conditions, it is again limited where exposure to hypoxic conditions was the focus. Available research is restricted

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further still when concerning the polymer acrylonitrile butadiene styrene (ABS). While ABS is often used in the formation of high end electronic equipment there appears to be limited research coverage compared to other common polymer types within the field of Fire Safety Engineering. Due to its common use and predictable burning behavior, there is a large collection of data related to polymethylmethacrylate (PMMA) in reduced oxygen environments.<sup>2-4</sup> Similarly, polyethylene (PE), polypropylene (PP), and polystyrene (PS) have also received both theoretical and experimental discussion in the works of researchers such as Tewarson et al.<sup>5,6</sup> and Kashiwagi.<sup>7</sup> ABS has been covered in previous works by others<sup>8</sup> but some of this work focuses on different types of polymer degradation and does not directly cover combustion reactions of ABS exposed to hypoxic conditions.<sup>9</sup> Other works have indeed covered the combustion of ABS in hypoxic conditions. However, they have discussed the material in a much broader context alongside many other materials and have not considered its behavioral changes when stored in a composite arrangement.<sup>10,11</sup>

The testing methodologies proposed in EN16750:2017<sup>12</sup> and VdS3527:2018<sup>13</sup> are intended to identify an ignition threshold for oxygen reduction systems (ORS). The ignition threshold is a target oxygen concentration that applies an approximate limiting oxygen concentration (LOC) of the materials contained within the protection zone and an additional safety margin (typically 0.75%-1.0% O<sub>2</sub> concentration). The application of the ignition threshold is in order to ensure the lowest concentration of oxygen necessary to prevent the ignition of stored materials. The test used to define the ignition threshold has been noted as being very material dependant and primarily for the testing of generic materials. Such limitations are of concern because of the likelihood that composite materials are contained within the protection zone.<sup>1</sup>

As a relatively new method of fire protection, the installation of ORSs is limited to 700 installations outside North America (as recorded in 2014).<sup>14</sup> As such, there is a limited number of case studies available in which to learn and improve system design. The limited number of installed systems is likely to slow the iterative evolution of system design. Due to the value, in both financial and societal terms, of the contents protected by ORSs, there is significant motivation to explore composite material behavior in hypoxic conditions. This motivation is further supported by the limited consideration of composite behavior within the experimental methodology of ORS design guidance as reported.<sup>1</sup> It is not the intention of this study to replicate the testing methodologies proposed for ORSs, but rather, to supplement the knowledge of material behavior in hypoxic conditions in order to improve the theoretical discussions used to guide design methodologies.

## 2 | THEORETICAL BASIS FOR THE USE OF OXYGEN CALORIMETRY

Oxygen consumption calorimetry is a long-established method of determining the heat release rate (HRR) of a material.<sup>15</sup> The HRR is determined by the principle of oxygen calorimetry, conceptually recognized by Thorton<sup>16</sup> and empirically confirmed by Parker<sup>17</sup> and Huggett,<sup>18</sup> where organic liquids and gases were shown to consume approximately the same mass of oxygen per net amount of heat that

is released (13.1(±5%) kJ per 1 g of O<sub>2</sub>). The method is widely used and standardization<sup>19</sup> has allowed for the production of replicable data assuring its wide-ranging use within the research community.<sup>20</sup>

Evidently, there are some critical design limitations to the standard cone calorimeter (CC) that limit its applicability to evaluate fire in hypoxic conditions, not least is its open design. It is the intent of the controlled atmosphere cone calorimeter (CACC) to maintain the operating capabilities of a typical cone calorimeter (CC) while being capable of operating at oxygen conditions which are different to those encountered in ambient environments. As noted by Leonard,<sup>21</sup> one of the primary differences between a typical CC and the controlled atmosphere alternative includes the introduction of a flow of N<sub>2</sub>/air to lower the volume fraction of O<sub>2</sub>. A CACC, therefore, introduces hypoxic conditions through vitiation of the environment by displacing oxygen with nitrogen rather than 'under-ventilation' where the flow of air is limited into the enclosure. This is important to note as ORSs also create hypoxic conditions through vitiation and key differences in material behavior have been observed between vitiated and under-ventilated conditions.<sup>22</sup>

Where a standard CC is used, ISO5660-1:2019 calculations can be used to determine the HRR. The study conducted made use of the alternative methods outlined by Werrel<sup>23</sup> where the HRR is calculated with a time-dependent mole fraction of oxygen in incoming air,  $X_{O_2}^{A^0}$ , rather than the constant value used in the typical CC method.

This calculation method, first proposed by Werrel,<sup>23</sup> describes how the use of time-dependent mole fractions in HRR calculations is due to the changing ratio of the mass flow rate into the exhaust duct during experiments using a CACC. This is due to the change in temperature, and therefore density, of the exhaust gases from the CACC chamber. The fan controlled extract system located in the CACC exhaust duct, typically running at a flow rate of 24 L/s (0.024 m<sup>3</sup>/s), has a constant volumetric flow rate while the fluctuating density within the CACC chamber will change the mass flow into the exhaust ductwork. This change in mass flow into the exhaust duct results in the changing rate of entrained air from the laboratory, mixing via the open chimney connection between the chamber and exhaust duct. Subsequently, there is a change in the ratio of the mass flow rate of the exhaust gases out of the chimney to the mass flow rate of the entrained ambient air. This process leads to dynamic baseline mole fractions rather than constant values found in the standard CC apparatus. This invalidates the assumption, made in standard cone calorimetry experiments, that mass flow rate into the exhaust flow stays constant and therefore baseline readings of effective mole fractions for constituent gases (used to calculate mass of oxygen consumed) stay the same during the experiment.

Other variations from ISO 5660-1:2019 are present, as considered by Werrel<sup>23</sup> in Equation (1) but have not been discussed further in this article. The changes to the calculation procedure have a significant impact on the results and have been shown to produce deviations in HRR results of approximately 30%.<sup>23</sup>

$$\dot{q} = E \cdot 1.10 \cdot \left( X_{O_2}^{A^0} \gamma - X_{O_2}^{A^s} (\gamma - 1) \right) \cdot C \sqrt{\frac{\Delta p}{T_e}} \left[ \frac{\phi - \frac{0.172(1-\phi)X_{CO}^A}{X_{O_2}^A}}{(1-\phi) + \phi \left( 1 + 0.5 \left( X_{O_2}^{A^0} \gamma - X_{O_2}^{A^s} (\gamma - 1) \right) \right)} \right] (1 - X_{H_2O}^s \tilde{\gamma}), \quad (1)$$

where:

$E$  = average value of the net heat release per unit mass of oxygen consumed (13.1 kJ/g<sub>O<sub>2</sub></sub>).

$\gamma$  = thermal expansion factor (-).

$\tilde{\gamma}$  = thermal changeable dilution factor (-).

$\Delta p$  = orifice meter pressure differential (Pa).

$T_e$  = absolute temperature of gas at the orifice meter (K).

$X_{CO}^A$  = measured mole fraction of carbon monoxide in exhaust gases (-).

$X_{O_2}^{A^S}$  = measured mole fraction of oxygen in the surrounding air (-).

$\phi$  = oxygen depletion factor (-).

$X_{O_2}^A$  = measured mole fraction of oxygen in the exhaust gases (-).

$X_{CO_2}^A$  = measured mole fraction of carbon dioxide in the exhaust gases (-).

$X_{CO_2}^{A^0}$  = measured mole fraction of carbon dioxide in incoming air (-).

$X_{H_2O}^{A^S}$  = measured mole fraction of water in the surrounding air (-).

With the oxygen depletion factor in this case defined as:

$$\phi = \frac{\left[ \left( X_{O_2}^{A^0} \gamma - X_{O_2}^{A^S} (\gamma - 1) \right) \left( 1 - X_{CO_2}^A - X_{CO}^A \right) \right] - \left[ X_{O_2}^A \left( 1 - X_{CO_2}^{A^S} \tilde{\gamma} \right) \right]}{\left( 1 - X_{O_2}^A - X_{CO_2}^A - X_{CO}^A \right) \left( X_{O_2}^{A^0} \gamma - X_{O_2}^{A^S} (\gamma - 1) \right)} \quad (2)$$

The methods described for determining heat release rate are one measure of burning behavior that is typically used in Fire Safety Engineering to quantify, evaluate, and compare material performance. These methods are not used in the testing regimes prescribed for ORSS, with instead, samples of 200 mm by 25 mm by 5 to 25 mm in reduced sized protected zones used to replicate the proposed system. The ignition threshold is subsequently determined through the use of observable performance to assign pass/fail criteria. However, oxygen consumption calorimetry been considered appropriate in the current study in order to create reproducible and comparable data for academic study beyond simple 'no-go/go' criteria used in practice.

### 3 | EXPERIMENTAL PROCEDURE

Tests were conducted using a bench scale CC, for ambient conditions, and using an 'open' CACC for hypoxic conditions. The open CACC was equipped with a 0.6 m chimney, designed according to ISO 13927:2015,<sup>24</sup> in order to prevent backflow of air and combustion of the volatiles interacting with ambient air after the chimney. All preparation and conditioning of samples were as per the methods described in ISO5660-1:2019. Refractory fiber blanket of 2 to 3 layers with one layer of refractory fiber millboard used for 5-mm samples. These layers were used as backing materials in order to ensure the surface of the sample was at the appropriate distance from the cone heater in all tests and to ensure limited heat losses through the sample holder during testing. The number of reruns of the same test was limited to three per test.

A total of 52-bench scale tests were conducted (26× on uniform samples of ABS and 26× on composite samples consisting of layered sheet of cardboard (×1 each approx. 2-mm thick), PE bubble wrap (×3 each approx. 1-mm thick), and ABS). The key variables within the tests were different imposed heat fluxes (25 and 50 kW/m<sup>2</sup>), fuel types (uniform and composite), and volume percent oxygen concentrations (20.95%, 17%, and 15%). The full experimental setup is shown in Figure 1.

Once the sample was introduced into the controlled atmosphere chamber, there was a period where it was necessary to reintroduce equilibrium into the chambers local atmosphere, which had been previously disrupted by opening the door to the chamber to insert the sample. Prior to the initiation of the experimental procedure, three timed trials were undertaken to determine the minimum required amount of time necessary to ensure oxygen levels had stabilized. This minimum time, determined to be 45 seconds, was used such that sample preheating was uniform and its effects limited. Due to the efforts made to minimize preheating, supported by the use of the CACC radiation protection shield, it is anticipated that preheating has had a minimal effect on results collected.

The necessary balance of N<sub>2</sub> to air flow rates required to achieve the intended oxygen concentration in the controlled atmosphere chamber were calculated as per the procedure given by equipment manufacturers (FTT Ltd.,<sup>25</sup>). The total flow rate of the incoming N<sub>2</sub>/air mixture was set to 140 L/min, which was the optimum flow rate achievable with the N<sub>2</sub> rate supplied from the available cylinders at both 17% and 15% O<sub>2</sub>. A higher flow rate is desirable to avoid stagnant zones within the chamber as described by Marquis.<sup>2</sup> The flow rate was used to achieve the desired hypoxic conditions of 17% and 15% O<sub>2</sub> although localized leakage introduced some variability during experiments. A variation of ±0.2% was considered acceptable and was monitored during the experimental run time.

It should be noted that the experimental work was planned and conducted in early 2020, prior to the release of ISO 5660-5:2020.<sup>26</sup> However, results can be considered broadly comparable as the methodology followed and apparatus design described in this work is

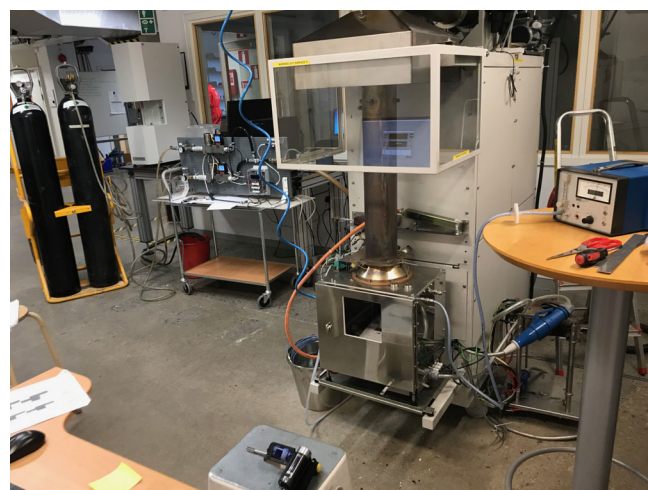


FIGURE 1 Experimental equipment set-up

similar to the guidance given in ISO 5660-5 with no notable deviations made.

## 4 | MATERIALS TESTED

As previously described, 26× on uniform samples of ABS and 26× on composite samples consisting of layered sheet of cardboard (×1 each approx. 2-mm thick), PE bubble wrap (×3 each approx. 1-mm thick) and ABS were used for testing. These materials as shown in Figure 2.

The ABS samples were sourced<sup>27</sup> and confirmed to contain the following distribution of base polymers; Styrene—58% to 63%, Acrylonitrile—20% to 25%, Butadiene—16% to 21%, Additives—1% to 4%. The bubble wrap sample was sourced<sup>28</sup> and was confirmed to be composed of polyethylene. Cardboard used for testing was sourced from spare cardboard packaging available within the laboratory. It is noted that, due to the wide-ranging potential of ORS, many different fuel arrangements could have been explored. ABS represents a popular thermoplastic for a wide range of uses because it is superior toughness and rigidity. These characteristics have been described as unusual for thermoplastics and explain the use of ABS for thousands of products in the housing, electronics, and automotive industry.<sup>29</sup> The cardboard and bubble wrap layers were chosen because cardboard was identified as the primary fuel load within ORS protected zones in a number of case studies.<sup>30,31</sup> One of these case studies featured the protection of a storage space containing high precision scales, which, from the product literature studied, often used ABS for the scale due to its robustness.<sup>32</sup>

It should be noted that the chosen composite layers are somewhat arbitrary. They do not represent a particular arrangement but instead a hypothetical arrangement based on examined case studies. While the CC has been said to adequately represent composite arrangements of 50 mm or less,<sup>33</sup> other researchers have reported complications in performance due to edge effects. Richardson and

Brooks<sup>34</sup> reported that composites with a surface layer of low burning propensity but an underlayer of high burning propensity performed in an unrepresentative fashion at the edges due to the early involvement of the composite underlayer. This effect was not observed during the experiments conducted as the surface layer was reported to ignite and burn much faster than the underlayer in this instance due to the material properties of the layers involved.

## 5 | RESULTS AND DISCUSSION

Experimental results are as shown in Tables 1 to 3 for materials at 50 kW/m<sup>2</sup>, uniform materials at 25 kW/m<sup>2</sup> and composite materials at 25 kW/m<sup>2</sup>, respectively. Result observations focus on the ignition and heat release rate results collected as well as discussions regarding the unique performance of the composite samples.

## 6 | IGNITION AND REIGNITION

### 6.1 | Influence of sample thickness

The material thickness was shown to influence the flameout time. This is predictable, where the samples did not self-extinguish and spent all available fuel, because of the increased fuel available in thicker samples. However, material thickness variations were observed to cause negligible changes in the ignition times of uniform ABS samples. It should be noted that sample thickness effects on time to ignition ( $t_{ig}$ ) are exclusively discussed within this section. Further discussions on the effect of hypoxic conditions on  $t_{ig}$  are discussed in a later section.

Varying the sample thickness in uniform samples in this instance, between 5 and 20 mm, does not appear to alter the material thermal gradient to such an extent that heat losses from the material surface are significant enough to prevent or delay igniting the sample due to the influence of the applied irradiance. This is in alignment of the findings of Berg and Lindgren,<sup>35</sup> and can be observed in Figure 3, where there is no clear trend toward increased  $t_{ig}$  values in thicker uniform samples.

To further investigate this, an approximation of whether the samples demonstrate semi-infinite behavior has been calculated using Equations (3) to (5) as found in Drysdale<sup>36</sup> in order to give an indication of whether a sample can be considered to contain thermal gradients that do not extend to ambient temperatures on the unexposed side. Where samples are semi-infinite, there is a thermal gradient within the length,  $L$ , at time,  $t$ , between the side heated by a uniform heat flux and the unexposed side (where ambient conditions are maintained).

Determining whether a sample is semi-infinite will not give confirmation on whether the sample is thermally thick, thin, or intermediate. This approximation will, however, give some understanding of heat penetration depths within the samples, and following from this, explain why no clear change in  $t_{ig}$  was observed.



**FIGURE 2** Samples tested (top to bottom, from left to right—composite 20-mm ABS, 10-mm ABS, 5-mm ABS, uniform 20-mm ABS, 10-mm ABS, 5-mm ABS)



**TABLE 1** Data from all materials tested at 50 kW/m<sup>2</sup>

Test group	Repeat test number	Oxygen concentration (vol%)	Material description	Sample density (kg/m <sup>3</sup> ) <sup>a</sup>	Peak HRR measurement (kW/m <sup>2</sup> ) <sup>b</sup>
1	1	20.95	ABS 5 mm	998	1089 ± 160
	2			1028	1068 ± 398
	3			922	1022 ± 202
2	1	20.95	Composite (5 mm ABS)	632	597 ± 43
	2			538	425 ± 49
	3			628	521 ± 47
3	1	20.95	ABS 20 mm	1006	1248 ± 382
	2			994	1134 ± 319
	3			1002	1120 ± 254
4	1	20.95	Composite (20 mm ABS)	835.2	444 ± 45
	2			829.2	367 ± 17
	3			838.8	357 ± 15
9	1	17.0	ABS 5 mm	1120	833 ± 355
	2				
	3				
10	1	17.0	Composite (5 mm ABS)	660	447 ± 410
	2				
	3				

<sup>a</sup>Composite sample densities have been calculated based on the total sample weight divided by the total thickness of the system and not on the basis of each independent layer.

<sup>b</sup>The error range of peak heat release rate measurements has been given by the range of values 25 seconds before and after the measured PHRR.

**TABLE 2** Data from uniform materials tested at 25 kW/m<sup>2</sup>

Test group	Repeat test number	Oxygen concentration (vol%)	Material description	Sample density (kg/m <sup>3</sup> )	Peak HRR measurement (kW/m <sup>2</sup> ) <sup>a</sup>
5	1	20.95	ABS 5 mm	1010	729 ± 101
	2			1462	716 ± 86
	3			1012	668 ± 64
6	1	20.95	ABS 10 mm	1013	645 ± 39
	2			1051	712 ± 110
	3			993	667 ± 44
11	1	17.0	ABS 5 mm	1314	561 ± 40
	2			1212	543 ± 61
	3			1280	557 ± 50
13	1	15.0	ABS 5 mm	1124	554 ± 104
	2			1010	515 ± 63
	3			1212	594 ± 58
15	1	17.0	ABS 10 mm	1092	524 ± 130
	2			1080	529 ± 47
	3			1097	501 ± 44
16	1	15.0	ABS 10 mm	1101	441 ± 47
	2			1074	436 ± 42
	3			1093	443 ± 48

<sup>a</sup>The error range of peak heat release rate measurements has been given by the range of values 25 seconds before and after the measured PHRR.

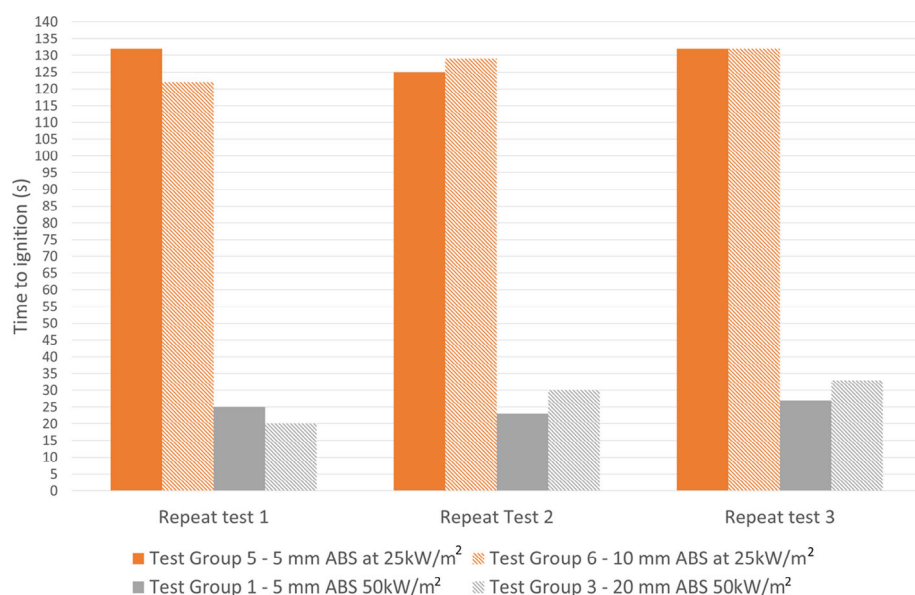
**TABLE 3** Data from composite materials tested at 25 kW/m<sup>2</sup>

Test group	Repeat test number	Oxygen concentration (vol%)	Material description	Sample density (kg/m <sup>3</sup> ) <sup>a</sup>	Peak HRR measurement (kW/m <sup>2</sup> ) <sup>b</sup>
7	1	20.95	Composite (10 mm ABS)	779.33	262 ± 17
	2			773.33	270 ± 47
	3			714	129 ± 4
8	1	20.95	Composite (5 mm ABS)	607	352 ± 23
	2			668	335 ± 19
	3			615	234 ± 15
12	1	17.0	Composite (5 mm ABS)	576	156 ± 16
	2			652	254 ± 37
	3			627	272 ± 34
14	1	15.0	Composite (5 mm ABS)	575	204 ± 27
	2			687	259 ± 29
	3			759	NA <sup>c</sup>
17	1	17.0	Composite (10 mm ABS)	838.66	314 ± 26
	2			800	316 ± 25
	3			822	308 ± 25
18	1	15.0	Composite (10 mm ABS)	816	NA <sup>c</sup>
	2			824.66	327 ± 19
	3			822	325 ± 19

<sup>a</sup>Composite sample densities have been calculated based on the total sample weight divided by the total thickness of the system and not on the basis of each independent layer.

<sup>b</sup>The error range of peak heat release rate measurements has been given by the range of values 25 seconds before and after the measured PHRR.

<sup>c</sup>Sample cardboard layer ignites but ignition of the ABS and bubble wrap underlayer does not occur.

**FIGURE 3** Time to ignition in ambient atmosphere tests between uniform sample thicknesses of 5 and 10/20 mm at irradiance levels of 25 and 50 kW/m<sup>2</sup>

An approximation of semi-infinite behavior in a one-dimensional slab can be calculated using Equations (3) and (4), where  $L$  is the thickness of the sample (m),  $t$  is the time of exposure to a uniform heat flux (s), and  $\alpha$  is the thermal diffusivity ( $k/\rho c$ ) (m<sup>2</sup>/s). Where Equation (4) equals approximately 2 then semi-infinite behavior can be assumed provided that Equation (3) is true.

$$L > 4\sqrt{(\alpha t)}, \quad (3)$$

$$\frac{L}{2\sqrt{(\alpha t)}} \approx 2. \quad (4)$$

It is also considered reasonable to assume semi-infinite behavior where Equation (5) is true<sup>36</sup>:

$$L > 2\sqrt{(\alpha t)}. \quad (5)$$

A value for the thermal diffusivity of ABS of  $1.65 \times 10^{-7} \text{ m}^2/\text{s}$  was used based on material properties found in the literature.<sup>37</sup> As the calculation methods are only intended for engineering approximation, the uniform samples of ABS have been considered since the presence of a rapidly forming soot and char layer would likely invalidate the approach. Approximations of semi-infinite behavior using calculations for a one-dimensional slab at a uniform heat flux are shown in Tables 4 and 5 for 25 and 50 kW/m<sup>2</sup>, respectively.

Results from Table 4 indicate that, at the time of ignition, the 5-mm sample is not semi-infinite, whereas the 10-mm sample is semi-infinite. It would be expected that a sample with a heated unexposed face would ignite faster than a comparable semi-infinite sample. However, it is noted that the value used to determine whether the 10-mm sample in Table 4 is semi-infinite is close to the boundary used by Equation (5) to indicate that the material is no longer semi-infinite. As this method is an approximation with high uncertainty the sample may not have ambient temperatures at the unexposed face at the time to ignition. If the 10-mm sample was indeed no longer semi-infinite, this would better explain the similar recorded  $t_{ig}$  between tested thicknesses at 25 kW/m<sup>2</sup>. If both thicknesses were not semi-infinite, it would suggest that heat penetration had reached the unexposed face of both the 5 and 10 mm samples, and subsequently, that heating of the entire body of the sample leads to ignition.

Results from Table 5 indicate that, at the time of ignition, markedly increased due to the increased irradiance, both samples are potentially semi-infinite. This would explain the similar  $t_{ig}$  values recorded in Figure 3 as the ignition criteria were primarily caused by irradiation onto the sample surface rather than the progressive heating of the entire body of the sample.

Analysis using engineering calculations suggests that differences in thermal inertia between samples at thicknesses ranging from 5 to 20 mm was not significant enough to effect time to ignition. This appears to be because the thicknesses examined did not affect whether  $t_{ig}$  was caused by a uniform rise in temperature of the entire sample body (as appears to be the case under 25 kW/m<sup>2</sup>) or the application of sufficiently high irradiance to the surface (as was the case for the 50 kW/m<sup>2</sup> sample).

The changes to ignition time in samples of different thicknesses are more significant in the composite samples, however, this is thought to be due to the effect of the soot and char residual layer rather than the thicknesses of the ABS, cardboard, and PE bubble wrap layers as discussed later.

## 6.2 | Influence of irradiance

Ignition times are shown to decrease under the exposure to higher levels of irradiation as has been shown in the work of Urbas,<sup>38</sup> where all but one fire rated timber material was found to ignite sooner where a higher level of irradiance within the CC was applied. This is clearly the case for uniform samples shown in Figure 4A,B. In Figure 4C,D, due to the low thermal capacity of the cardboard top layer, both levels of applied irradiation are sufficient to induce ignition almost immediately. However, the secondary peak in heat release rate, where the cardboard and bubble wrap layers are burnt away and the underlayer of ABS ignites, still indicates the effect of irradiance as both sets of tests with an irradiance of 50 kW/m<sup>2</sup> show a faster secondary rise in HRR. There are indications, however, of the increased influence of material thickness for samples comprising of composite layers. In Figure 4D, the secondary peak behavior of the 5 mm ABS underlayer samples now demonstrate steeper rates of rise in HRR, which were exactly the same as the 20 mm ABS underlayer in the uniform samples shown in Figure 4B. These will be of further discussion where the impact of the composite layer is examined.

## 6.3 | Influence of oxygen concentration

Ignition, as illustrated in Figures 5 and 6, was measured through observation and then validated through reviewing video recordings of the experiments. The error bars in Figure 5 show the minimum and maximum  $t_{ig}$  values, collected over three repeat tests, for the corresponding test group. There is a conflict within reviewed literature regarding the relationship between the  $t_{ig}$  and oxygen concentration. There are studies indicating a strong correlation<sup>39</sup> and others that observed negligible effect where  $t_{ig}$  and critical heat flux were found to be independent of oxygen concentration in fire rated and

**TABLE 4** Semi-infinite behavior of ABS at tested thicknesses at 25 kW/m<sup>2</sup>

Test material	Sample thickness (m)	Thermal diffusivity from literature (m <sup>2</sup> /s)	Time to ignition (s) <sup>a</sup>	Equation (3)	Equation (4) <sup>b</sup>	Equation (5)	Notes
ABS	0.005	$1.65 \times 10^{-737}$	129	0.018		0.009	Equations (3) + (4) inconclusive. Equation (5) indicates non semi-infinite
	0.01	$1.65 \times 10^{-737}$	127	0.018		0.009	Equations (3) + (4) inconclusive. Equation (5) indicates semi-infinite

<sup>a</sup>Time to ignition has been taken as the mean value recorded over three repeat tests in uniform samples at the relevant thickness.

<sup>b</sup>Where the preliminary criteria required in Equation (3) are not met, Equation (4) has not been recorded.

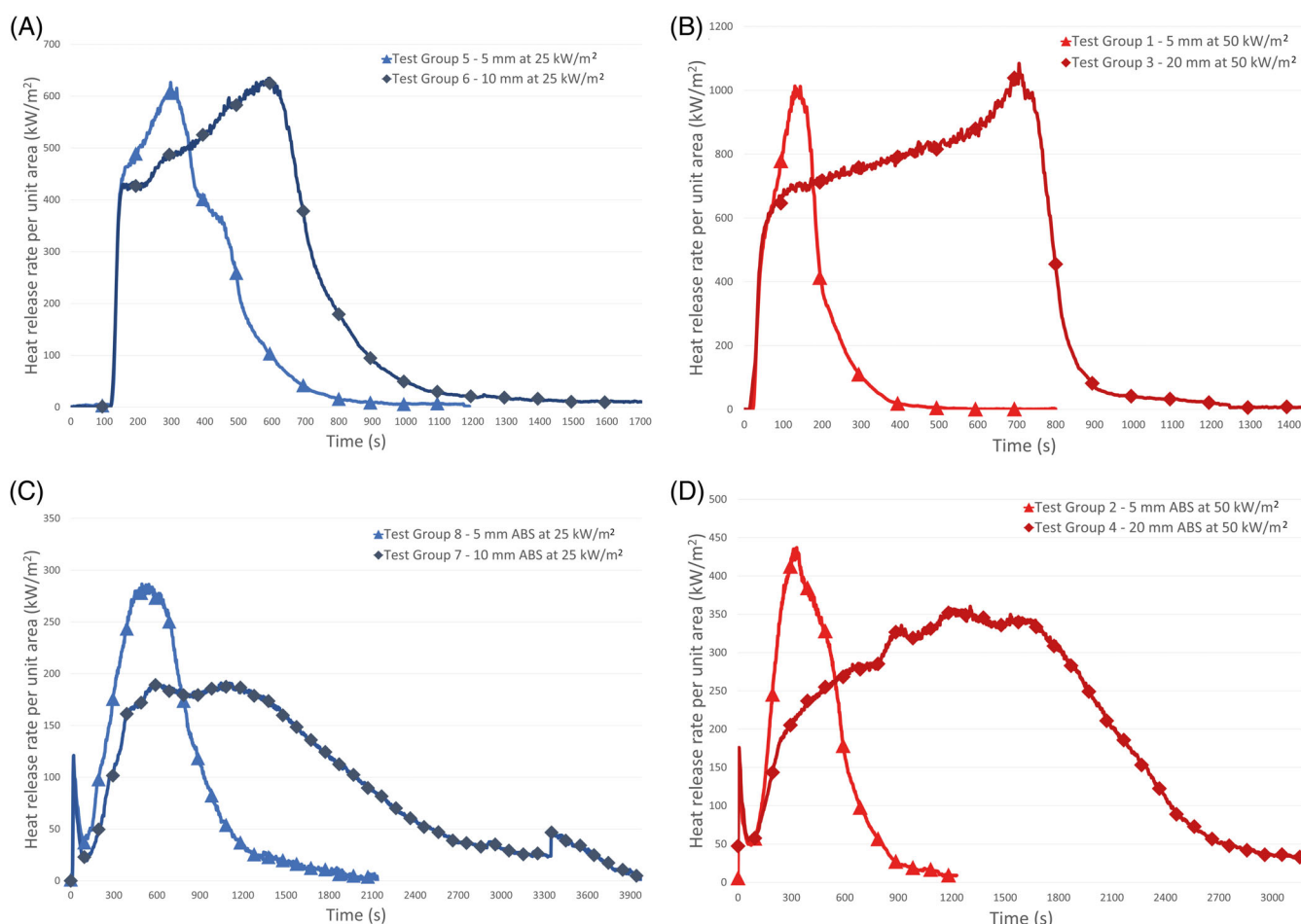


**TABLE 5** Semi-infinite behavior of ABS at tested thicknesses at 50 kW/m<sup>2</sup>

Test material	Sample thickness (m)	Thermal diffusivity from literature (m <sup>2</sup> /s)	Time to ignition (s) <sup>a</sup>	Equation (3)	Equation (4) <sup>b</sup>	Equation (5)	Notes
ABS	0.005	$1.65 \times 10^{-737}$	25	0.0081		0.004	Equations (3) + (4) inconclusive. Equation (5) indicates semi-infinite
	0.02	$1.65 \times 10^{-737}$	27	0.0084	4.7	0.004	Equations (3) + (4) inconclusive. Equation (5) indicates semi-infinite

<sup>a</sup>Time to ignition has been taken as the mean value recorded over three repeat tests in uniform samples at the relevant thickness.

<sup>b</sup>Where the preliminary criteria required in Equation (3) are not met, Equation (4) has not been recorded.



**FIGURE 4** Average of heat release rate per unit area (kW/m<sup>2</sup>) values for uniform and composite samples at 20.95% O<sub>2</sub> over three repeat tests (A, Uniform samples exposed to 25 kW/m<sup>2</sup> at 5 and 10 mm; B, Uniform samples exposed to 50 kW/m<sup>2</sup> at 5 and 20 mm; C, Composite samples exposed to 25 kW/m<sup>2</sup> at 5 and 10 mm; D, Uniform samples exposed to 50 kW/m<sup>2</sup> at 5 and 20 mm)

nonfire rated wood.<sup>40</sup> There is some risk that the use of the inlet flow rate into the CACC may influence the  $t_{ig}$  of samples and that this may be misinterpreted as being due to the oxygen concentration imposed rather than the effects of apparatus dependency.<sup>2</sup> Observations illustrated in Figure 6 appear to indicate a slight increase in  $t_{ig}$  between ambient and hypoxic conditions for uniform samples of both 5- and 10-mm thick. However, the increase in  $t_{ig}$  between 17% and 15% is slightly less conclusive as there is no pattern emerging to allow for a

definitive conclusion from the data. While the data indicate that the  $t_{ig}$  increased slightly when oxygen concentrations were reduced, further testing is necessary to determine the true extent of this effect and the dependency on apparatus design. The most notable cases where oxygen concentration influences  $t_{ig}$  are all cases where there is a composite arrangement as shown in Figure 5. This is, in part, due to the changes in burning behavior due to the combination of composite layering and hypoxic conditions as discussed in a later section.

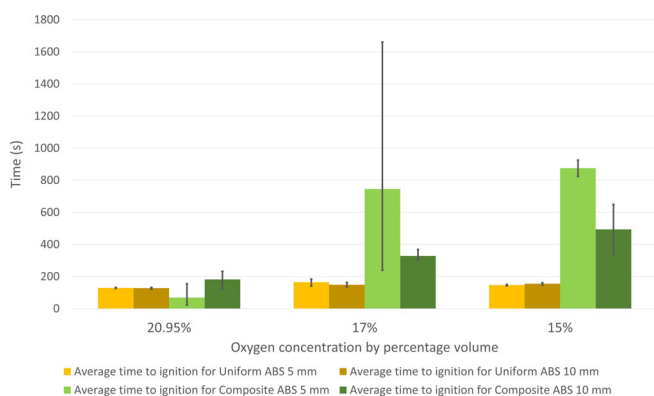
## 6.4 | Influence of composite form

The composite form was shown to have a significant impact on ignition times in multiple ways. First, as shown in Figure 4, the introduction of composite layers disrupted the clear relationships between ignition, sample thickness, and imposed irradiance. The general trends outlined in Figure 4 display similar trends as summarized by Schartel and Hull<sup>41</sup> with the changes introduced by the composite layers similar to the HRR vs time graphs typically associated with char/residue forming materials. The introduction of composite layers changed the ignition times, caused flame extinction in some samples, and introduced the importance of sample reignition. All of which changed the predictability of ignition behavior in the uniform samples where the thicknesses had minimal impact and applied irradiance was the determining factor. It should also be noted that the lower oxygen concentration tests, which included a composite layer, had a higher likelihood of failing to ignite or self-extinguishing than the equivalent uniform sample. For example, while none of 12 uniform samples in

hypoxic conditions self-extinguished all 12 composite samples in hypoxic conditions self-extinguished.

Further evidence of the impact of composite layers on material behavior is shown in Figure 5. It should be noted that the times taken for 'ignition' in this instance are for 'sustained ignition' and therefore represent the point at which the ABS layer ignites rather than the flashing observed in the bubble wrap and cardboard layer. Rather than tens of seconds difference, as observed in uniform samples, the  $t_{ig}$  difference between tests in ambient and hypoxic conditions is now hundreds of seconds. This demonstrates that there are multiple determining factors that contribute to the impact of hypoxic conditions on  $t_{ig}$ , potentially explaining the conflicting reports on this subject, and that composite layers may contribute to altering the impact of oxygen concentration on  $t_{ig}$ .

It may be that, for composite samples, the demonstrated effect of oxygen concentration on ignition time is not a direct effect, but rather, due to the secondary effects of incomplete combustion of the top layers. Reignition time was observed to increase significantly due to the combined effects of hypoxic conditions and composite layering. This combined effect was also in part due to applied irradiance, horizontal orientation of the sample and the formation of residual layers. While these effects are somewhat dependent on apparatus, results highlight the impact of confounding variables in influencing the ignitability of samples.

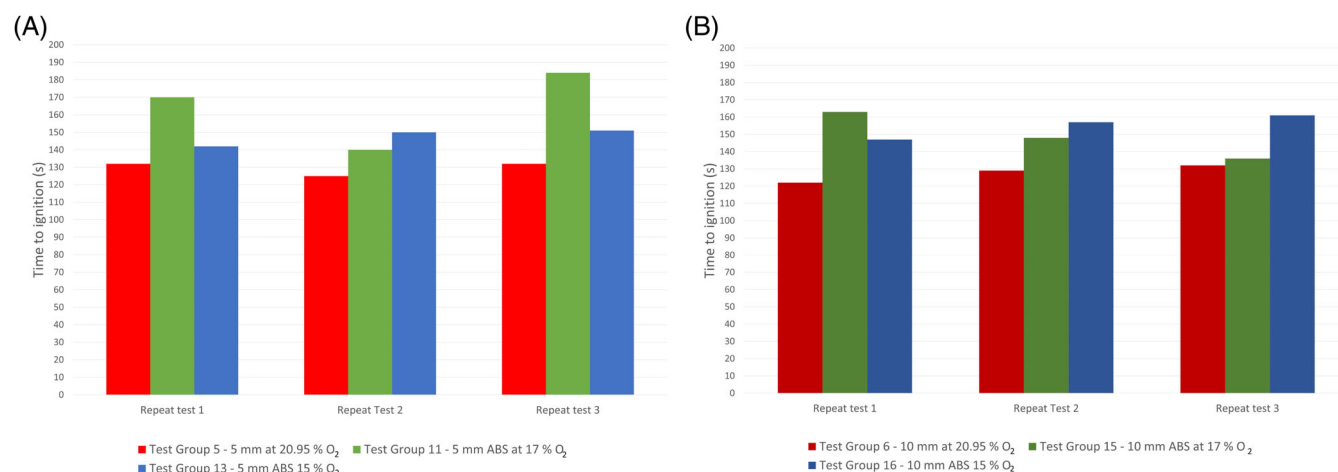


**FIGURE 5** Average time at which flaming was observed to occur in uniform and composite samples at an irradiance of 25 kW/m<sup>2</sup> over three repeat tests

## 7 | HEAT RELEASE RATE

### 7.1 | Influence of sample thickness

The 5-mm sample rapidly achieved its peak heat release rate per unit area (HRRPUA) value before immediately displaying similarly rapid HRRPUA reduction. The thicker, 20-mm sample displayed an extended period of relative equilibrium. The observed impact of thickness, and therefore to some extent, thermal thickness, on burning behavior was apparent. Tests demonstrate the effect of sample



**FIGURE 6** Time to ignition in samples exposed to a heat flux of 25 kW/m<sup>2</sup> comparing various atmospheric conditions and thicknesses in uniform ABS samples A: Uniform ABS with a thickness of 5 mm. B: Uniform ABS with a thickness of 10 mm

thickness on HRR in the shape and peak values that indicate typically 'thinner' and 'thicker' material behavior as summarized by Schartel and Hull.<sup>41</sup> Examples from the collected data, illustrating thinner and thicker behavior, respectively, have been given in Figures 7 and 8.

## 7.2 | Influence of irradiance

Irradiance was shown to be the largest contributing factor to heat release rates. Higher levels of irradiance caused greater peaks in heat release rate consistently. This was particularly true for the uniform samples shown in Figure 4A,B but also the composite samples as shown in Figure 4C,D.

## 7.3 | Influence of oxygen concentration

HRRPUA were generally shown to be lower when hypoxic conditions were introduced. This was true for all uniform samples. Reducing the oxygen volume fraction was also shown to alter the relationship between material thickness and peak HRRPUA as is shown in Figure 9.

Thicker, uniform samples demonstrate a greater reduction in peak HRRPUA when oxygen levels are reduced with a more gradual incline in heat release rate growth over time. As the HRRPUA lowers the material thickness of the sample has a greater significance on the overall transference of heat internally within the sample. For uniform samples at ambient conditions, the comparable HRRPUA peaks for different sample thicknesses suggest that the thermal inertia of these samples has a negligible effect on peak heat release. Following from this, it is also evident that thermal inertia, and therefore sample thickness, becomes more critical to peak HRRPUA as the oxygen volume fraction is lowered.

The combined effect of hypoxic conditions and increased material thickness in the uniform samples are not demonstrated in composite samples. This is as shown in Figure 10 where the gradient of the HRRPUA rise observes no notable difference when thickness is

increased. The cause of this is discussed further in later discussions regarding confounding variable effects of hypoxic conditions and composite form. The behavior of Sample 12.1 in Figure 10, where hypoxic conditions prevent the ignition of the sample until the lapse of a significant delay in time, is interesting as it demonstrates the effects of late stage reignition on HRR. During the delay in reignition, there would have been sample decomposition, which would have taken place due to the imposed irradiance. The sudden reignition of the sample either suggests a cracking in the soot and char residual layer or the interaction of the decomposing sample with the sample holder backing materials causing a reduction in heat losses, and subsequently, a rise in pyrolysis temperature such that the critical heat flux for ignition was exceeded. Both phenomena have been recorded as typical causes for a late stage growth in HRR in materials with a char layer<sup>41</sup> and may be responsible for material behavior changes leading to delayed reignition in this instance.

## 7.4 | Composite form

Consistently lower peak HRRPUA were observed between composite experiments and the respective uniform equivalent. The reduced heat release output caused by composite layers further enhanced the likelihood of extinction due to the reduced transference of volatiles to the flaming region.

Results from the composite sample data suggest that material thickness does have a greater impact on the self-extinguishment of the samples than was observed in the uniform material tests. Composite samples of 10-mm ABS were more likely to self-extinguish than composite samples with 5-mm ABS. While both sets of samples self-extinguished under hypoxic conditions only, the thicker 10-mm ABS sample also self-extinguished in all ambient tests. This suggests that material thickness was more critical to self-extinguishment in a composite arrangement. It is hypothesized that the soot and char residual layer of the composite sample increased the criticality of heat losses in the protected virgin fuel to the point that sustained combustion ceased where thicker samples included a composite layer.

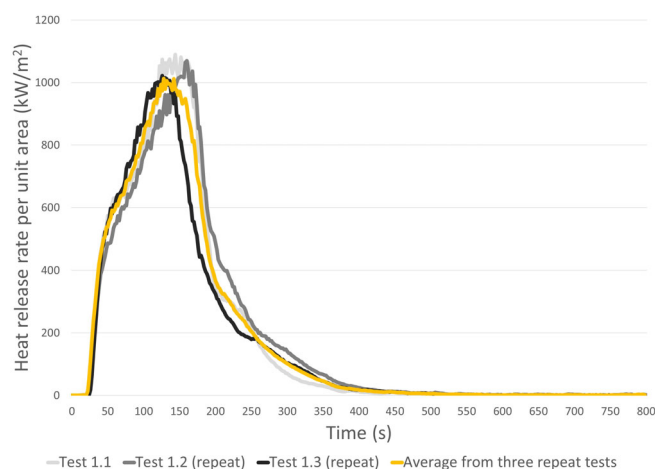


FIGURE 7 Test 1—Uniform ABS 5 mm 50 kW/m<sup>2</sup> at 20.95% O<sub>2</sub>

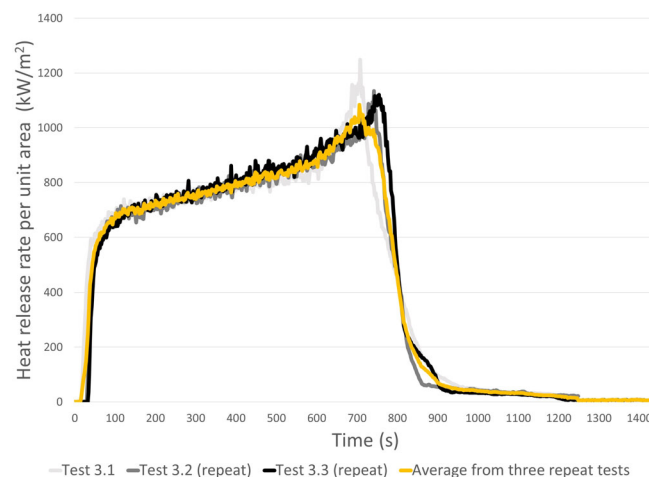
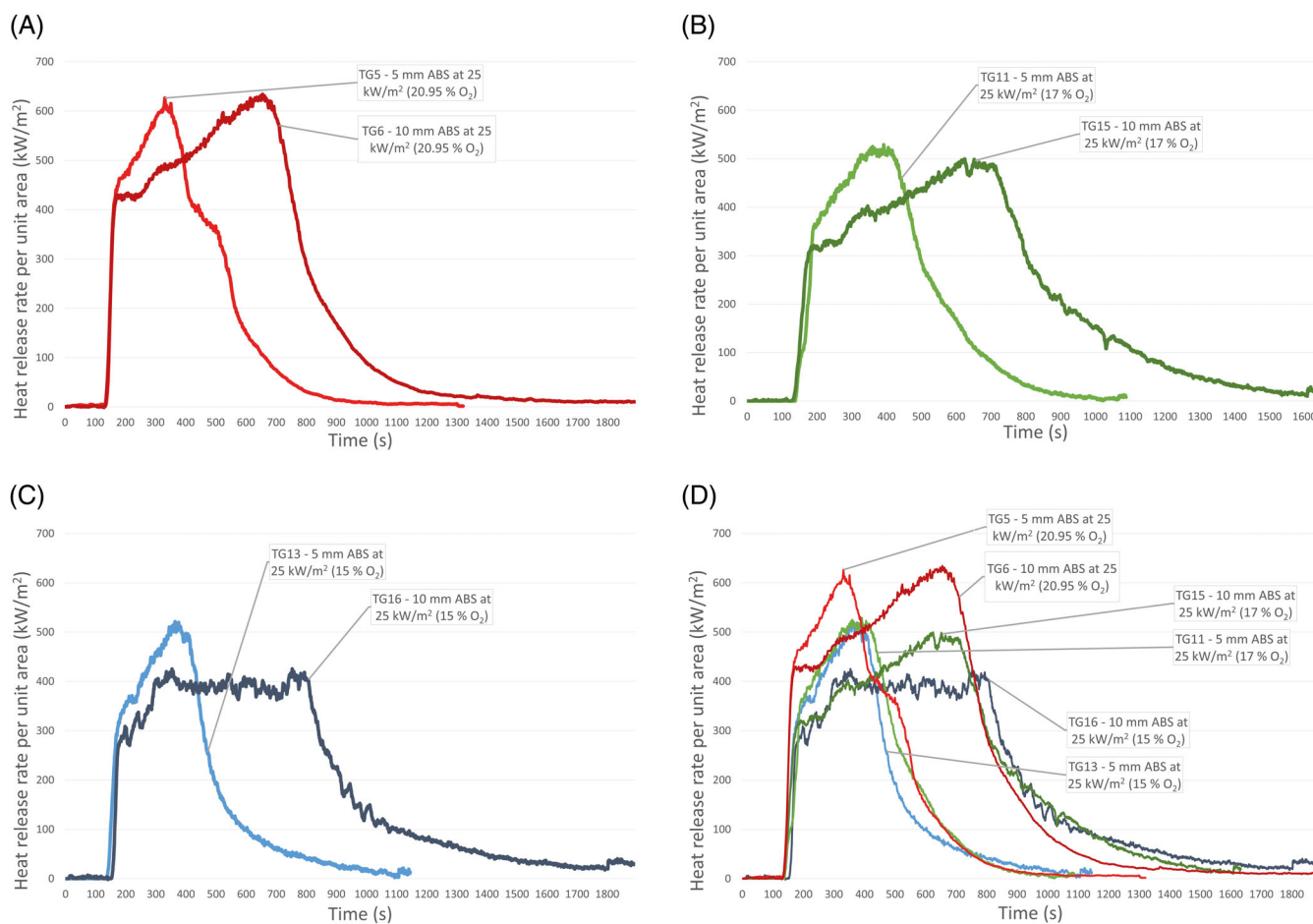


FIGURE 8 Test 3—Uniform ABS 20 mm 50 kW/m<sup>2</sup> at 20.95% O<sub>2</sub>



**FIGURE 9** Average of heat release rate per unit area ( $\text{kW/m}^2$ ) values for uniform ABS samples over three repeat tests (A, 20.95%  $\text{O}_2$ ; B, 17%  $\text{O}_2$ ; C, 15%  $\text{O}_2$ ; D, Combined)

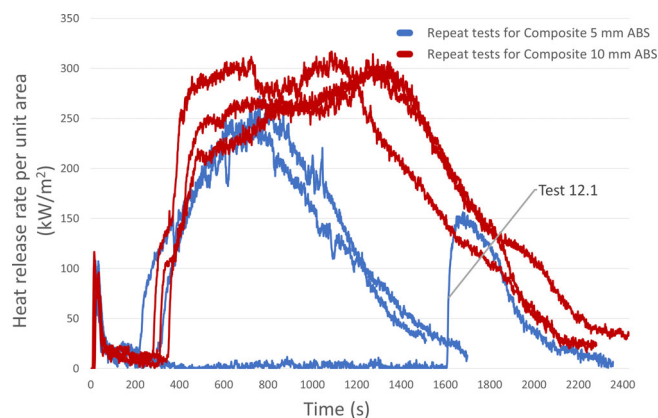
## 8 | BURNING BEHAVIOR OBSERVATIONS

### 8.1 | Oxygen concentration and composite form

The variation of sample behavior between tests demonstrated that there was a link between the impact of composite layers and the oxygen concentration introduced. The change in behaviors observed where hypoxic conditions were introduced to uniform samples were different than observations made for composite samples in significant ways.

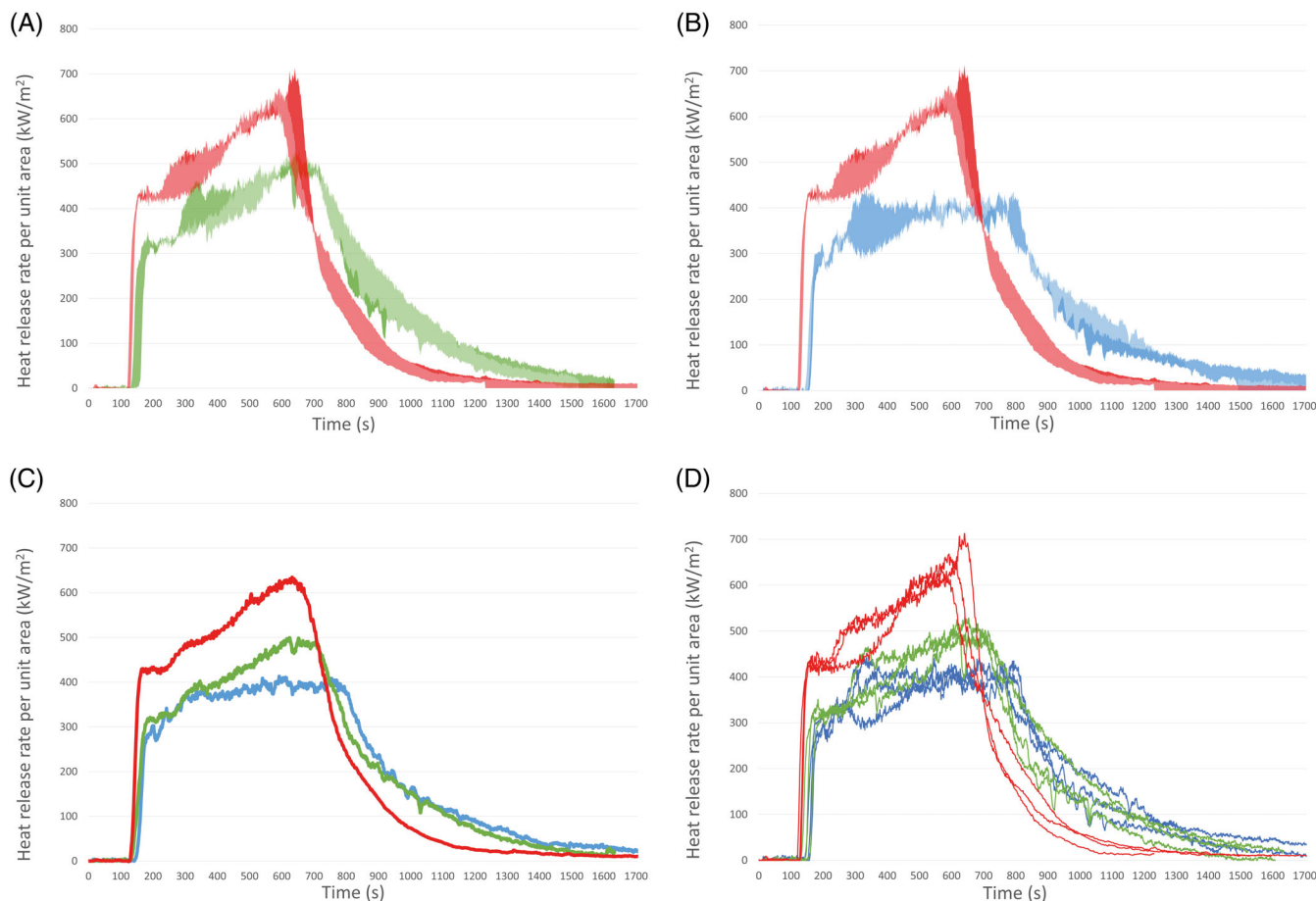
As can be observed in Figure 9, the relationship between material thickness and the exposure to lower volume fractions of oxygen for the uniform sample set displays a trend where, as the volume fraction of oxygen is reduced, the HRRPUA peak for samples is reduced also. These reductions of HRRPUA were observed to be greater for thicker samples. The uniform samples observed correlation between heat release rates, material thicknesses, and oxygen volume fractions. The relationship between HRRPUA and oxygen concentration in uniform samples, in this case from Tests 6, 15, and 16, has been shown in Figure 11.

Such trends are not observed in Figure 12 where the influence of material thickness can no longer be discerned by the shape of the HRRPUA curve. It is evident that, with the introduction of composite



**FIGURE 10** Heat release rate per unit area ( $\text{kW/m}^2$ ) values for composite ABS samples at 17%  $\text{O}_2$  (5- and 10-mm ABS)

layers, there is no longer a clear trend that allows for relative predictability in burning behavior. A particularly striking observation is that, in some cases, hypoxic tests are shown to have a higher peak HRR than the ambient oxygen volume fraction tests. For example, to compare composite results at 25  $\text{kW/m}^2$  with 10-mm ABS underlayer one



**FIGURE 11** Heat release rate per unit area ( $\text{kW}/\text{m}^2$ ) for uniform 10 mm ABS samples exposed to  $25 \text{ kW}/\text{m}^2$  irradiance (red = 20.95%  $\text{O}_2$ , green = 17%  $\text{O}_2$ , blue = 15%  $\text{O}_2$ ). A, Range of HRRPUA over three repeat tests for ambient and 17%  $\text{O}_2$  hypoxic conditions. B, Range of HRRPUA over three repeat tests for ambient and 15%  $\text{O}_2$  hypoxic conditions. C, Average HRRPUA over three repeat tests. D, All HRRPUA for three repeat experiments in test groups 6, 15, and 16

can see that five out of six of the hypoxic tests had higher peak heat release rates than any of the three ambient tests.

Broadly, there appears to be two primary contradictions between the composite and uniform tests:

- Typical HRRPUA indications of thicker/thinner behavior, based on the thickness of the sample, are no longer clearly observed.
- Typical indications of the effect of hypoxic conditions on HRRPUA are no longer clearly observed.

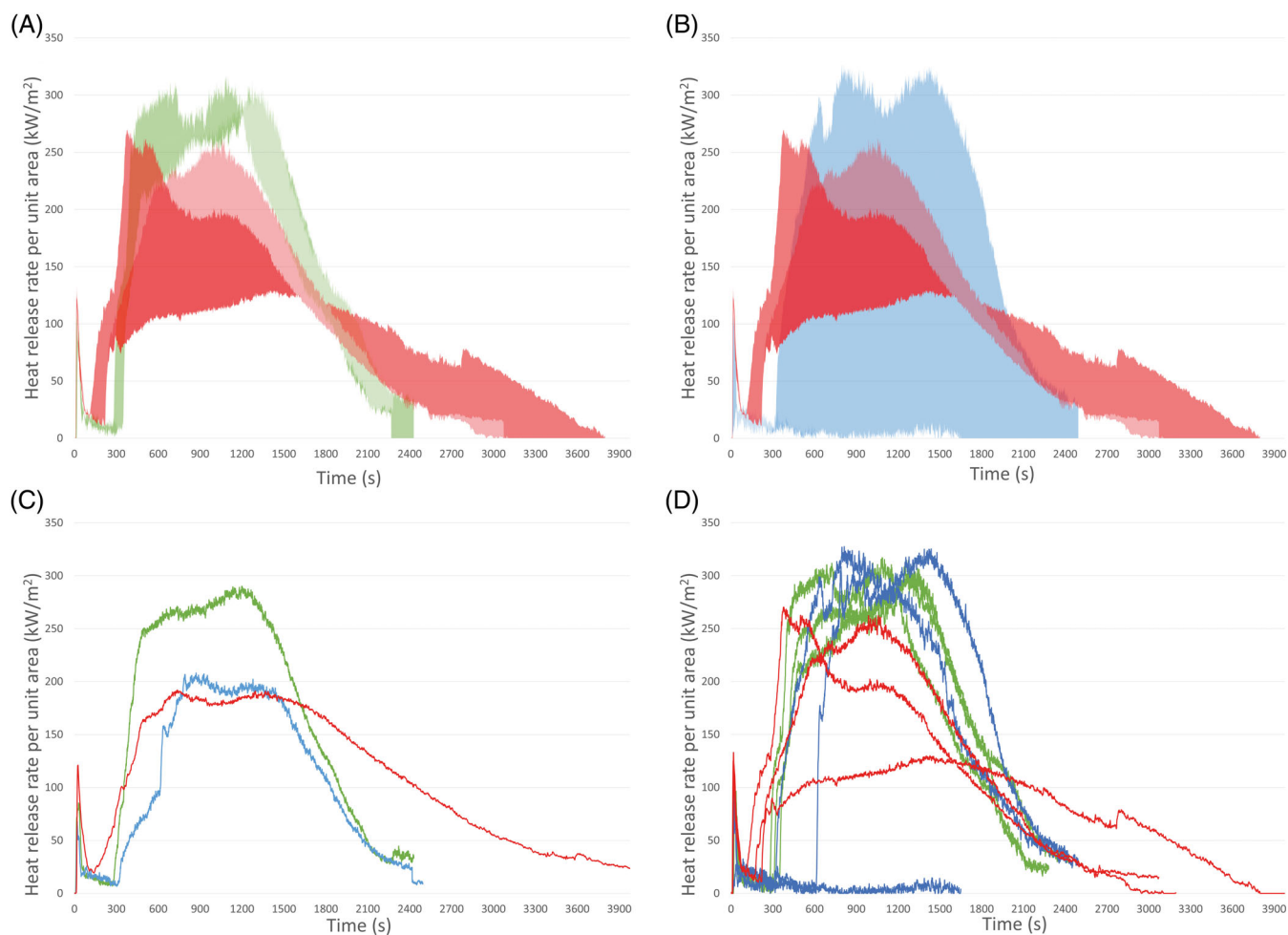
It is hypothesized that there are two primary causes for the alteration of typical behavior in composite samples exposed to hypoxic conditions in the tests conducted. These are:

- The initial burning of the cardboard layer, its combustion efficiency during this stage and the coverage of the subsequent soot and char residual layer created once the cardboard layer has ignited.
- The time between self-extinguishment of the initial flaming cardboard, caused by the development of the protective soot and char residual layer, and the reignition of the ABS due to the

reintroduction of the spark igniter. Over this time, preheating occurs due to the application of an external heat flux. The delay in reignition time, that is, the time between initial extinction and reignition, has been shown in Figure 13. This shows a trend of increased delay to reignition where hypoxic conditions are used in the CACC.

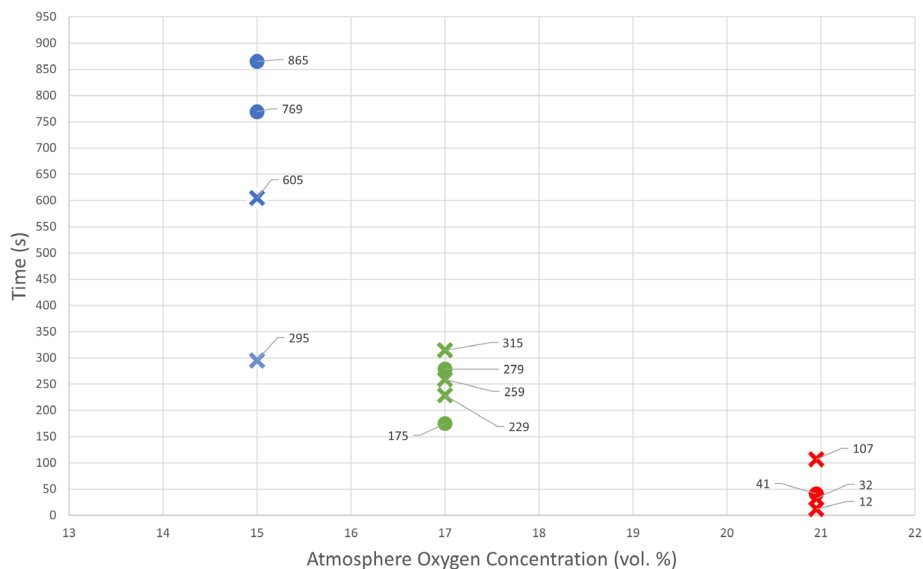
The formation of a top layer of soot and char over the ABS prevented thin samples from rapidly heating such that a short peak of HRRPUA area was observed as expected from thin samples. This is not the case for the composite test where there was 20.95% oxygen (ambient conditions) due to the burning efficiency of the cardboard layer such that an insufficient soot and char layer formed over the ABS layer below. In this instance, the peak HRRPUA for the thinner sample was greater than the thicker equivalent test, as expected and as observed in uniform tests.

The heating of the sample between self-extinguishment and reignition, prolonged by the hypoxic conditions, prevented thicker composite samples from demonstrating typical behavior as thermal equilibrium was reached throughout the sample over the time leading up to reignition.



**FIGURE 12** Heat release rate per unit area (kW/m<sup>2</sup>) values for composite 10-mm ABS samples (15% vs 17% vs 20.95%) exposed to 25 kW/m<sup>2</sup> irradiance (red = 20.95% O<sub>2</sub>, green = 17% O<sub>2</sub>, blue = 15% O<sub>2</sub>). A, Range of HRRPUA over three repeat tests for ambient and 17% O<sub>2</sub> hypoxic conditions. B, Range of HRRPUA over three repeat tests for ambient and 15% O<sub>2</sub> hypoxic conditions. C, Average HRRPUA over three repeat tests. D, All HRRPUA for three repeat experiments in test groups 7, 17, and 18

**FIGURE 13** Delay in reignition under different atmospheric conditions in composite samples (circles are composite repeat tests with 5-mm ABS, whereas crosses are composite repeat tests with 10-mm ABS)





The results appear to indicate that the anticipated observations between hypoxic conditions and HRR, as well as more general burning behavior, are broadly true for uniform materials. Reducing the oxygen volume fraction can be shown to reduce flaming and subsequently lower peak values for HRR and increase the likelihood of self-extinction. However, the assumed behaviors do not directly translate when applied to composite samples where applied in the horizontal orientation in bench scale testing due to the influence of other factors involved in the burning process.

## 9 | GENERAL TRENDS AND OBSERVATIONS OF MATERIAL PERFORMANCE

A number of general trends were recognized during testing of the composite samples. These have been summarized as:

- While the thicknesses of the uniform samples did not appear to change the likelihood of the material to ignite, extinguish, or reignite the introduction of composite layers did cause samples with thicker ABS layers to extinguish more readily. Composite samples of 10 mm ABS were less likely to ignite or reignite than composite samples where the ABS layer was 5 mm. The impact of material layer thickness appeared to be greater for composite samples than for uniform ones.
- The likelihood of the uniform samples to fail to ignite under hypoxic conditions did not increase. However, thicker uniform samples did demonstrate a greater reduction in peak HRRPUA as the volume fraction of oxygen was lowered than was observed for the thinner sample groups. It is likely that this was due to the change in ratio of 'heat released in flaming combustion' and 'heat losses into the material' when hypoxic conditions are introduced. Greater heat losses into a thicker material become a more relevant factor where the heat production rate from flaming combustion is reduced.
- Composite tests demonstrated that behavioral interactions between different layers of the sample could lead to significantly conflicting results. In numerous tests, the typical thicker/thinner behavior patterns shown in uniform samples were no longer apparent due to the effect of the protective soot and char layer over the ABS as well as the subsequent increase in preheating through exposure to an external irradiance. Test data even suggests that, in certain conditions, the HRRPUA under hypoxic conditions could be higher than otherwise expected in ambient conditions.
- The added complexity introduced by the impact of composite layers also suggests that the required minimum number of repeat tests should be scrutinized by the laboratory team where composite arrangements are of interest.
- Comparisons in  $t_{ig}$  appear to show that hypoxic conditions did increase the time to ignition when compared to ambient test data. However, the change from 17% and 15%  $O_2$  had a negligible effect on  $t_{ig}$  for uniform samples. Differences in ignition times were much greater in composite samples. Composite samples saw  $t_{ig}$  increase by hundreds of seconds, compared to tens of seconds for uniform

samples, where hypoxic conditions were introduced. There was also significant variability introduced to  $t_{ig}$  results where composite layers were present.

## 10 | CONCLUSIONS

The study conducted has focused on the comparison of ambient and hypoxic atmospheres in bench scale tested samples. The tests carried out aimed to provisionally identify characteristic behaviors that differentiate material performance of uniform/generic materials and those stored in composite arrangements when exposed to an ignition source. The primary aim of the work undertaken was to supplement academic knowledge of material behavior in hypoxic conditions in order to improve the theoretical discussions used to guide ORS design methodologies.

One of the challenges of investigating the effects of composite systems featuring materials in sequence is the inherent reliance of the system on not only the materials themselves, but also their interaction with each other through fixings and the sequence in which they make contact with an ignition source. The work undertaken has focused on one such composite system in order to investigate the interaction of composite systems and hypoxic conditions. The results may not necessarily reflect the performance of any other composite arrangement other than those tested. Further investigation of other common storage materials, and air-filled components within them, is desired to further appreciate their impact on system performance under hypoxic conditions.

Experimental results have identified a number of examples where the influence of composite layers has resulted in significantly different outcomes for material burning behavior than otherwise observed in uniform materials. This was demonstrated to even produce instances where the HRR was greater in hypoxic conditions than in ambient conditions for some composite samples. The interaction between hypoxic conditions and material layers demonstrates that testing materials in isolation cannot be assumed to yield results that correspond to materials where placed in series.

The results highlight important, interacting factors that should be considered when designing testing methodologies for fire prevention or protection systems. Large-scale experiments should be conducted that explore the combined influence of composite layers and hypoxic conditions on material performance with a focus on how this corresponds to the anticipated fire scenarios envisaged for the fire prevention or protection systems of interest. There is a desire within the fire safety community to investigate correlation between large- and small-scale hypoxic test methodologies in order to provide results that are simultaneously able to appreciate complex interactions between experimental variables while maintaining cost efficiency. Comparisons between small- and large-scale tests will produce data that can potentially prove useful in offering more robust testing methodologies than the 'pass/fail' tests currently used in hypoxic system design but are unlikely to allow the prediction of real world fire events.

## ACKNOWLEDGEMENTS

The authors would like to thank the material suppliers, lab staff, and associated organizations that supported the work over a challenging period interrupted by the onset of the Covid-19 pandemic as it spreads into Europe. Robert John Bray would also like to thank the International Masters in Fire Safety Engineering (IMFSE) program for the support and guidance that assisted with the completion of the project.

## DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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**How to cite this article:** Bray RJ, Barton J, Madsen D, van Hees P. Burning material behaviour in hypoxic environments: An experimental study examining a representative storage arrangement of acrylonitrile butadiene styrene, polyethylene bubble wrap, and cardboard layers as a composite system. *Fire and Materials*. 2021;1–16. <https://doi.org/10.1002/fam.3002>